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Magnetic properties of L1₀ FePt and FePt:Ag nanocluster films

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A sputtering gas-aggregation technique has been used to prepare FePt and FePt:Ag nanocluster films. The cluster size was controlled in a range from 3 to 6 nm. FePt cluster films were directly deposited onto Si substrate; FePt:Ag cluster films were fabricated by depositing a FePt cluster layer between a Ag underlayer and overlayer. Nanostructure and magnetic properties of the samples were characterized by x-ray diffraction, transmission electron microscopy, and magnetometry. The high magnetic anisotropy L1₀ fct phase was realized in the films annealed at a temperature of 550 °C and above. The orientation of clusters is random. The coercivity increases with an increase of annealing temperature; high in-plane and out-of-plane coercivities, exceeding 10 kOe, were achieved in both FePt and FePt:Ag cluster films after annealing. For FePt:Ag films, the coercivity increases with Ag underlayer thickness, t_{Ag} , and reaches about 17 kOe at room temperature for $t_{\text{Ag}}=5$ nm after annealing at 650 °C for 10 min. The high coercivity is closely correlated with the degree of L1₀ ordering and nanostructure of the films. © 2003 American Institute of Physics.
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I. INTRODUCTION

In recent years there has been considerable interest in FePt nanoparticles with the high magnetic anisotropy L1₀ ordered structure for extremely high-density magnetic recording (EHDR) media.^{1–4} The requirement of noise reduction for extremely high magnetic recording density, e.g., from 100 Gb/in² to 1 Tb/in², imposes the need for grain size being reduced below 10 nm with extremely uniform size distribution.⁵ As a consequence, such small particles of the standard CoCrPtX type, will cause the thermal instability of magnetic recording because of superparamagnetic limitation. The equiatomic ordered face-centered-tetragonal (fct) phase FePt alloy has a high anisotropy constant, about 7×10^7 erg/cm³, which is of crucial importance for EHDR media with a grain size below 10 nm, because the reduction of grain size can be balanced by large anisotropy based on the thermal stability factor $K_u V/k_B T$. The high $K_u V$ value can create a barrier to thermally activated switching of the magnetization. Most FePt nanoparticles were prepared with normal magnetron sputtering or electron-beam-evaporation techniques.^{1–4} New techniques such as chemical synthesis and self-assembly methods have been developed recently to obtain FePt nanoparticles with very uniform particle size and narrow size distribution.⁶

In this article FePt clusters are produced with a gas-aggregation technique, in which magnetron sputtering is employed in the source.⁷ This cluster-deposition technique can produce a very large range of mean cluster sizes from 200 to

15 000 atoms per cluster with high fluxes. Thus it is suitable for preparation of FePt cluster-based materials. The success of this application may provide an alternative method for fabrication of extremely high-density magnetic recording

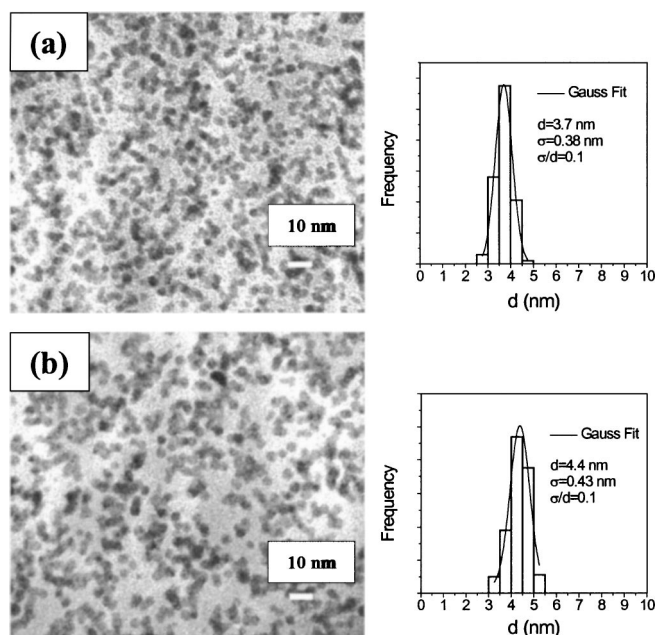


FIG. 1. TEM images of FePt nanoclusters: (a) deposited at power 60 W with Ar/He gas flow 250/250 sccm; and (b) at power 100 W with Ar/He gas flow 300/200 sccm. The right side shows the corresponding cluster size distribution.

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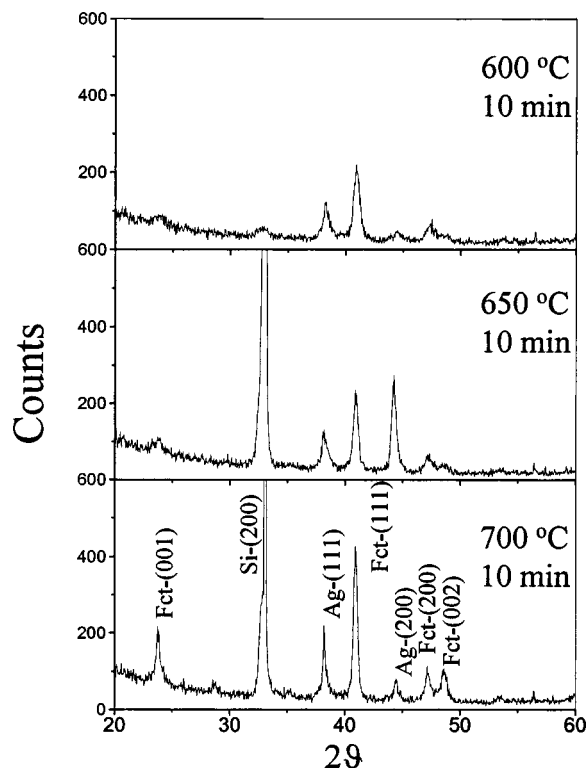


FIG. 2. XRD ($\theta-2\theta$) scans of Ag(2 nm)/FePt(12 nm)/Ag(2 nm)/Si nanocluster films annealed at different temperature for 10 min.

media or permit the exploration of other novel properties of nanocluster films.

II. EXPERIMENT

FePt and FePt:Ag cluster films are prepared using a cluster-deposition system with a sputtering gas-aggregation source. FePt clusters were formed in a LN₂ cooled chamber filled with high pressure Ar-He gas. Detail of the cluster deposition will be published elsewhere.⁸ The base pressure of the cluster-forming chamber was $<1 \times 10^{-7}$ Torr. During deposition, the chamber was cooled below -120°C , and the Ar-He pressure was about 5×10^{-1} Torr. This high pressure restricted the glow discharge region to a few millimeters from the target, allowing the sputtered FePt atoms to grow into clusters in the cooled Ar-He gas atmosphere by repeated collisions with the carrier gas.

FePt nanoclusters are directly deposited onto Si or 7059 glass substrates; FePt:Ag cluster films are deposited with a FePt cluster layer between a Ag underlayer and overlayer. The thickness of the FePt layer was varied from 12 to 15 nm. The as-deposited films were annealed at various temperatures in a rapid thermal annealing (RTA) furnace in an Ar gas flow. Nanostructure of the films was examined with a Rigaku x-ray diffractometer (XRD) using Cu $K\alpha$ radiation and a JEOL 2010 transmission electron microscope (TEM). Magnetic properties were measured by a superconducting quantum interference device (SQUID).

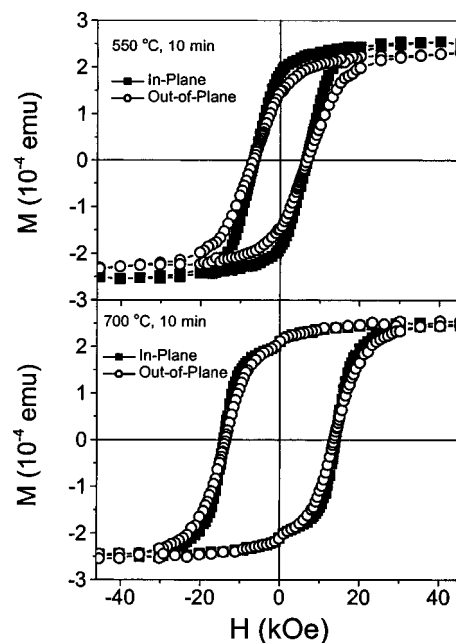


FIG. 3. Hysteresis loops of FePt(15 nm)/Si nanocluster film annealed at 550 and 700 °C for 10 min.

III. RESULTS AND DISCUSSION

Figure 1 shows the TEM images of FePt nanoclusters as deposited on carbon-coated films supported by Cu grids. Shown on the right-hand side is a corresponding cluster size distribution. Spherical clusters with an average size of 3.7 nm (standard deviation $\sigma=0.38$ nm) were prepared with sputtering power of 60 W and Ar/He gas flow of 250/250 [Fig. 1(a)]; while clusters with size of 4.4 nm ($\sigma=0.43$ nm) were prepared with 100 W and gas flow of 300/200 sccm [Fig. 1(b)]. The cluster size can be controlled in a range from 3 to 6 nm by adjusting the sputtering power and Ar-He gas flow and gas pressure. The size distribution with $\sigma/d=0.1$ is fairly good, and can be further improved if desired by applying a mass selector. In the following sections the cluster films were prepared using power of 100 W. XRD measurement confirmed the ordering of L1₀ structure by postannealing. Figure 2 shows the XRD ($\theta-2\theta$) scans of Ag(2 nm)/FePt (12 nm)/Ag(2 nm)/Si cluster film annealed at

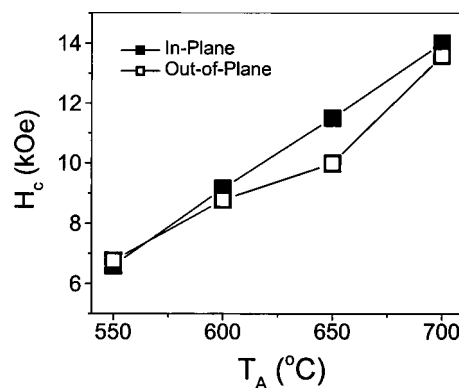


FIG. 4. Effect of annealing temperature on coercivity of FePt(15 nm)/Si nanocluster films. Annealing time: 10 min.

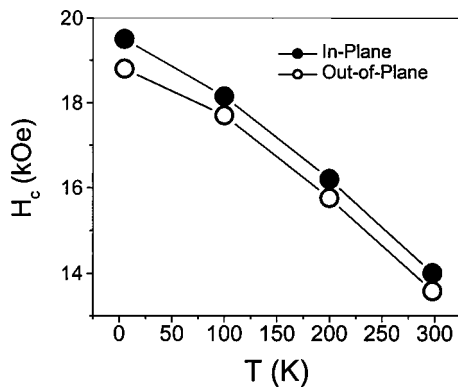


FIG. 5. Coercivity dependence on temperature for FePt(15 nm)/Si cluster film annealed at 700 °C for 10 min.

600, 650, and 700 °C for 10 min, respectively. The as-deposited film showed a broad FePt fcc (111) peak and Ag fcc (111) and (200) peaks. After annealing at 600 °C for 10 min, the FePt fct (001) and (002) peaks appeared, and the (200) and (002) peaks overlapped, indicating that the $L1_0$ ordering developed in the clusters, although was not completed. After annealing at 650 °C for 10 min, the (200) and (002) peaks were well separated, and the intensities of the fct peaks increased with increase of annealing temperature, indicating the $L1_0$ ordering was almost completed. In all the above films, the orientation of the fct FePt clusters is random.

Magnetic properties of the cluster films were measured by SQUID. Figure 3 shows the room temperature hysteresis loops of the FePt(15 nm)/Si films annealed at 550 and 700 °C for 10 min. Both in-plane ($H \parallel$ film plane) and perpendicular ($H \perp$ film plane) loops are presented. The perpendicular coercivity is almost the same as the in-plane coercivity, indicating that the easy axis of clusters is distributed randomly. This result is consistent with that obtained by XRD measurement (see Fig. 2). The degree of ordering depends on the annealing temperatures; large coercivity of the cluster films originated from the large K_u of the fct phase. Figure 4 shows the effect of annealing temperature on coercivity of the cluster films. The coercivity exceeded 6 kOe after annealing at 550 °C, indicating that most of FePt clusters have transformed into fct phase at this stage of annealing. The coercivity increases with increase of annealing temperature linearly, and high in-plane and out-of-plane coercivities, approaching 14 kOe, have been achieved after annealing at 700 °C for 10 min. In order to determine the thermal effect on coercivity, the hysteresis loops of FePt cluster films were measured at low temperature down to 5 K. Figure 5 shows the coercivity dependence on temperature for FePt (15 nm)/Si cluster film from 5 to 300 K. The film was annealed at 700 °C for 10 min. The coercivity decreases with increase of measuring temperature, dropping from 19 kOe at 5 K to

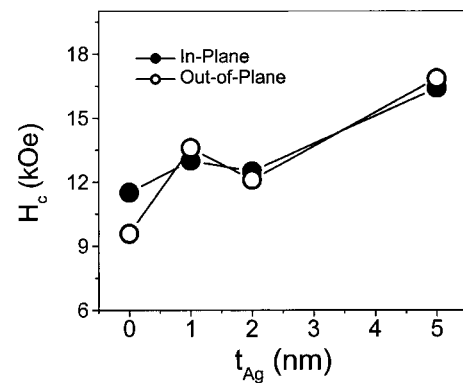


FIG. 6. Effect of Ag underlayer thickness on coercivity of Ag/FePt/Ag (t nm)/Si films annealed at 650 °C for 10 min.

about 14 kOe at 300 K. This is caused by a contribution of intrinsic temperature dependence of the anisotropy and magnetization, and thermal activation effects.⁹ Coercivities of Ag/FePt/Ag films show a similar tendency with respect to annealing temperature and thermal effect. Figure 6 shows the effect of the Ag underlayer thickness on the coercivity of the Ag/FePt/Ag cluster films annealed at 650 °C for 10 min. The coercivity increased with increase of Ag underlayer thickness, and reached about 17 kOe at room temperature as Ag underlayer thickness equals about 5 nm. Compared with the coercivity of 14 kOe for FePt/Si film annealed at 700 °C (see Fig. 4), this result suggests that addition of Ag underlayer can lower $L1_0$ ordering temperature and increase the coercivity of the cluster films significantly. It is likely that this results from the partial interdiffusion of the Ag underlayers and overlayers between and into the FePt clusters.

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